# Deepwater Horizon – Data Validation Report
# PAH & Alkylated Homologs by GC/MS-SIM

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|---|---|---|
| **SDG:** 1005014 | **Matrix:** Water | **Number of Samples:** 15 |
| **Laboratory:** Alpha Analytical Services | | **Method/SOP:** Alpha SOP/0-008 (Issue 6) |
| **Validation Level:** Stage 4 Validation | | **Validation Criteria Table:** MC252-PAH, Rev. 0 |
| **Date of Report:** July 20, 2010 | | **Approved for Release:** |

*Refer to the **SAMPLE INDEX** for a list of validated samples.*

*Refer to the **DATA VALIDATION PLAN** for validation approach, Criteria Tables, qualifier and reason code definitions.*

The quality control (QC) elements that were reviewed are listed below.

| | | | |
|---|---|---|---|
| 1 | Data Package Completeness | 1 | Sample Duplicate Analysis |
| √ | Verification of EDD to Hardcopy Data Package | 2 | Blank Spike/Blank Spike Duplicate Sample Analyses |
| 1 | Chain-of-Custody and Sample Receipt | 1 | Reference Material Analysis |
| √ | Holding Times | √ | Internal Standards |
| √ | Instrument Tuning | √ | Detection Limits |
| √ | Initial Calibration | √ | Target Analyte List |
| √ | Initial Calibration Verification | 2 | Compound Quantitation |
| √ | Continuing Calibration | √ | Compound Identification/Spectral Match (Stage 4 only) |
| √ | Method Blank Analysis | 1 | Mass Discrimination (Stage 4 only) |
| √ | Surrogate Compound Recovery | 1 | Calculation Verification (Stage 4 only) |

**√ Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.**

**1 Quality control results are discussed below, but no data were qualified.**

**2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed in this Data Validation Report.**

## Overall Assessment

Data were estimated for blank spike/blank spike duplicate recovery outliers and matrix interferences. Data were also estimated to indicate analytes that were not included in the calibration mixture.

All data, as qualified, are acceptable for use.

## Data Package Completeness

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory sample receipt form was not present in the report for this SDG; however, the six coolers received were logged in to two SDG, 1005013 and 1005014. The sample receipt form was present in SDG 1005013 and no further action was taken. The laboratory submitted all other required deliverables.

## Verification of EDD to Hardcopy Data Package

Sample results and related quality control data were received in both an electronic and hardcopy format. Electronic data were verified against the laboratory report; no errors were found.

## Chain-of-Custody and Sample Receipt

Sample identification (ID) numbers listed on the chain-of-custody record are consistent with the sample ID reported in the laboratory electronic data deliverable (EDD) and hardcopy data package.

With the exceptions noted below, samples were received within the advisory temperature range of 2° to 6°C (Analytical Quality Assurance Plan (AQAP), Section 3.1).

The laboratory received several of the sample coolers with temperatures above the advisory control limits, with the high temperature at 9.9°C. These outliers did not impact data quality and no qualifiers were required.

## Holding Times

Samples were analyzed within the holding time specified in the Analytical AQAP, Section 3.1, and documented in the Validation Criteria Table.

## Instrument Tuning

Instrument tuning was performed at the required frequency and met all criteria as specified in the referenced laboratory analytical SOP.

## Initial Calibration

Initial calibration (ICAL) standards were analyzed at the required frequency and the percent difference (%RSD) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table.

## Initial Calibration Verification

Initial calibration verification (ICV) standards were analyzed required frequency and the percent recovery (%R) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table. The ICV was a separate standard prepared from a second source.

## Continuing Calibration

Continuing calibration (CCAL) standards were analyzed at the required frequency and the percent difference (%D) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table.

## Method Blank Analysis

Method blanks were analyzed at the appropriate frequency. No analytes were detected in the associated method blank.

## Surrogate Compound Recovery

The percent recovery (%R) values for surrogates were within the control limits of 40% – 120% (control limits for d12-perylene are 10% – 120%).

## Sample Duplicate Analysis

Sample duplicate analysis was not performed on this SDG. Precision was evaluated using the blank spike duplicate analysis.

## Blank Spike/Blank Spike Duplicate Sample Analyses

One set of blank spike/blank spike duplicate (BS/BSD) samples (for each analytical batch of 20 or fewer samples) was extracted and analyzed. The percent recovery (%R) and relative percent difference (RPD) values were calculated and evaluated.

With the exceptions noted below, the %R values were within the criteria of 50% – 125%.

| Analyte | BS %R | BSD %R | Potential Bias |
|---|---|---|---|
| Benzo(a)anthracene | 130 | 126 | High |
| Benzo(a)pyrene | 146 | 143 | High |
| Chrysene/Triphenylene | 129 | 128 | High |
| Naphthalene | -- | 136 | High |

No action was taken for the naphthalene BSD outlier because the blank spike recovery was acceptable. Benzo(a)pyrene was not detected in the field samples; therefore no action was necessary. The positive results for benzo(a)anthracene, chrysene/triphenylene, and alkylated chrysenes were estimated (J-10).

All RPD values were less than the control limit of $\leq$30%.

## Reference Material Analysis

The reference material SRM 1582 was analyzed. The recovery of benzo(a)pyrene exceeded the upper control limit. Benzo(a)pyrene was not detected in the field samples; therefore no action was necessary.

An aliquot of North Slope Crude was analyzed. All recovery values were within the laboratory defined criteria of 65% – 135%.

## Internal Standards

The percent recovery (%R) values for internal standards (IS) were within the control limits of 50% – 200% of the area in the associated CCAL.

## Compound Quantitation

The laboratory applied a J-flag to all results between the quantitation limit (QL) and the method detection limit (MDL). During validation, results reported at less than the MDL were qualified as "found" (F).

A number of individual analytes (such as 4-methyldibenzothiophene) were not included in the calibration mixture. These analytes use the response factor of a designated 'parent' compound (such as dibenzothiophene) for calculation of the analyte concentration. Because the response factor of the parent compound can be significantly different than that of the individual analyte, all positive results for non-calibrated individual analytes are estimated (J-24).

The laboratory flagged the results for several analytes with a "G", indicating matrix interference that affected quantitation. These "G" flagged results were estimated (J-14).

## Mass Discrimination

The ratio for the raw areas of benzo[g,h,i]perylene to phenanthrene (calculated for the ICAL and CCAL) was $\geq 0.70$.

## Calculation Verification

Stage 4 validation was performed on this SDG. No transcription or calculation errors were found.

# Attachment 1: Sample Index - SDG 1005014
# PAH & Alkylated Homologs by GC/MS-SIM

| Sample ID | Lab ID | Date Collected |
|---|---|---|
| JF.4KM.DEEP.WD.20100512.N088 | 1005014-01 | 5/12/2010 |
| JF.8KM.BLANK.DIWD,20100512.N075 | 1005014-02 | 5/12/2010 |
| JF.2KM.MIX75.WD.20100513.N136 | 1005014-03 | 5/13/2010 |
| JF.2KM.MIX30.WD.20100513.N140 | 1005014-04 | 5/13/2010 |
| JF.2KM.400.WD.DUP.20100513.N160 | 1005014-05 | 5/13/2010 |
| JF.2KM.DEEP.WD.20100513.N124 | 1005014-06 | 5/13/2010 |
| JF.2KM.SURF.WD.20100513.N148 | 1005014-07 | 5/13/2010 |
| JF.8KM.MID.WD.20100512.N051 | 1005014-08 | 5/12/2010 |
| JF.8KM.SURF.WD.20100512.N071 | 1005014-09 | 5/12/2010 |
| JF.8KM.MIX30.WD.20100512.N067 | 1005014-10 | 5/12/2010 |
| JF.4KM.SURF.WD.20100512.N112 | 1005014-11 | 5/12/2010 |
| JF.2KM.LEESURF.WD.20100513.N168 | 1005014-12 | 5/13/2010 |
| JF.2KM.135FT.WD.20100513.N132 | 1005014-13 | 5/13/2010 |
| JF.2KM.BLANK.DIWD.20100513.N164 | 1005014-14 | 5/13/2010 |
| JF.2KM.400.WD.20100513.N152 | 1005014-15 | 5/13/2010 |